

Microstructural investigation of M-type BaFe₁₂O₁₉ hexaferrites using electron backscatter diffraction (EBSD)

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The analysis of the achieved texture is of great importance for ferrite materials, either bulk or thin films. The commonly employed techniques like X-ray pole figures do, however, not reach the required resolution to resolve fine details within the grain structure. Therefore, a technique with a relatively high spatial resolution would be required. The recently developed electron backscatter diffraction (EBSD) technique, which works within a scanning electron microscope, enables a spatially resolved study (a resolution of about 20 nm is possible on perfectly prepared surfaces of oxidic materials [1,2]) of the crystallographic orientations by means of recording of Kikuchi patterns. A good surface polishing/cleaning is essential for this analysis, as the method requires an undisturbed surface area for a high image quality. This information is recorded to each measured Kikuchi pattern, together with a parameter describing the quality of indexation. To each pattern, the three Eulerian angles determine the crystallographic orientation [3]. From this information, orientation maps and local pole figures can be constructed, enabling further a detailed analysis of grain boundaries.

In this contribution, we present an EBSD analysis of two bulk M-type hexaferrite BaFe₁₂O₁₉ (Ba-ferrite) samples, considering especially the quality of the surface preparation (IQ maps), the grain orientation (IPF maps) and the grain aspect ratio analysis. The samples were prepared by conventional ceramic processing [4]. Two samples, N2 and N3, which differ in the magnetic alignment process, were chosen for the present analysis. The surface preparation process of the bulk ferrites is a delicate task, as for a high spatial resolution a mechanical polishing down to 40 nm colloidal silica is required, which is then followed by a thermal etching or by Ar-ion polishing. Figure 1 (a) presents the obtained Kikuchi patterns after the ion-polishing step with Ar-ions (5 keV, 5 min), and Fig. 1 (b) shows the image quality (IQ)-maps (upper row) and the inverse pole figure (IPF) maps (lower row) of both samples under study.

Figure 2 presents EBSD-determined grain shape aspect ratio maps and plots. Here, the grain shape aspect ratio, g_r , is defined as the minor axis of an ellipse divided by its major axis, so a grain with a needle-like shape (i.e., a high aspect ratio) has $g_r \sim 0.1 \dots 0.2$. Only the grains which are fully included within the selected area take part in this analysis; the other grains are represented in white. Furthermore, as the crystallographic orientation of each individual grain is known, also the exact orientation of the major and minor axes of the ellipses are always well defined. Therefore, this type of plot enables a correct analysis as compared to e.g. optical methods. Figure 2 reveals clear differences between the two samples where sample N2 shows a large maximum at $g_r \sim 0.6$.

The spatially highly resolved EBSD analysis contributes, therefore, to choose an optimum processing condition for a given compound. The information on the individual grain orientation provides important input to understand the variation of the magnetic properties of

ferrite samples, and helps to improve the ferrite materials even further. Additionally, even grains down to several tens of nanometres can be directly analysed. In summary, we have performed a grain shape aspect ratio analysis by means of EBSD. Knowing the individual orientation of each grain enables an exact analysis of the grain shape aspect ratio also for tiny grains, so that exact results can be obtained. Especially for materials with a large anisotropy, this analysis may provide important information for the processing.

References

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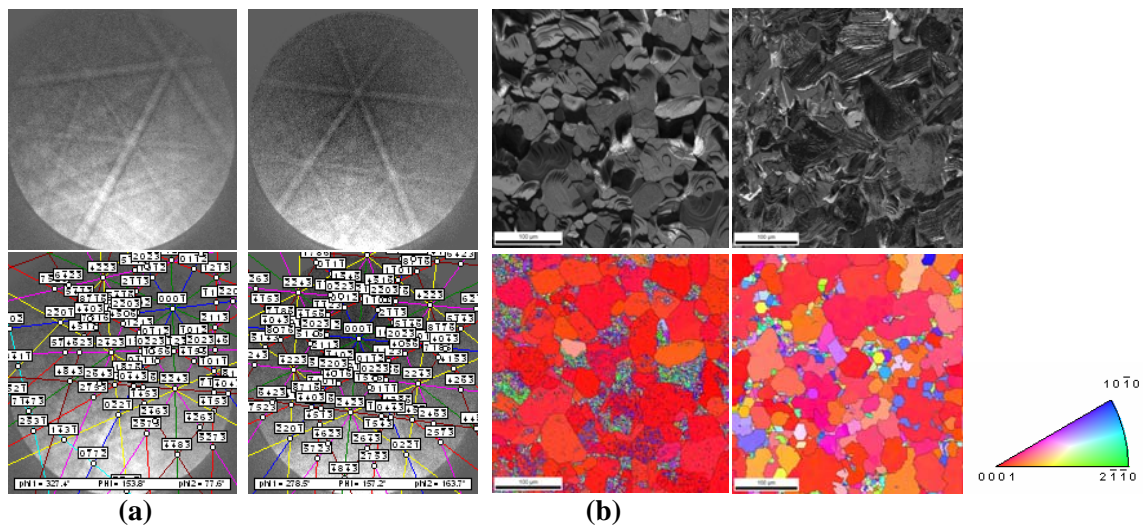


Fig.1 (a) Kikuchi patterns and indexation for $\text{BaFe}_{12}\text{O}_{19}$ ferrites after ion-polishing; (b) IQ- and IPF-(001) maps for samples N2 and N3. The colour code for the IPF maps is given in the stereographic triangle.

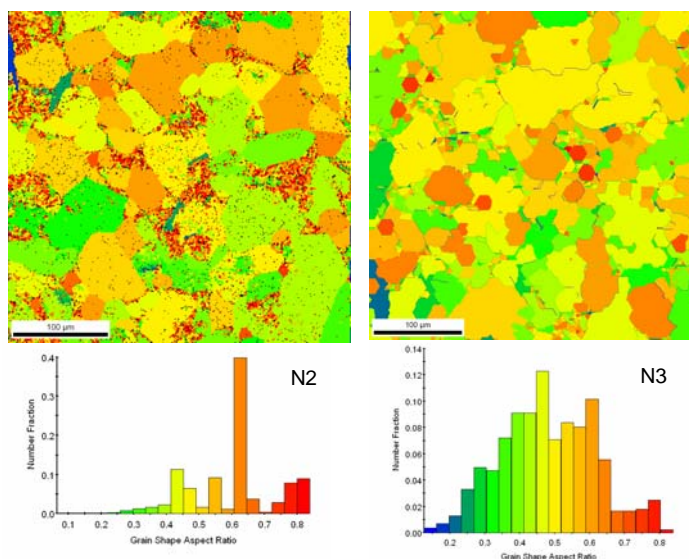


Fig.2 Grain shape aspect ratio plots and histograms (color code) for samples N2 and N3. The colour code for the maps is given in the plots below.